MICROWAVE ASSISTED GREEN SYNTHESIS OF ZNO AND AG-DOPED ZNO NANOPARTICLES USING *AEGLE MARMELOS* LEAF EXTRACT

SYNOPSIS

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1. Introduction

Nowdays nanoscience and nanotechnology have been established one of the extreme dynamic investigation zones in physics, chemistry, engineering and biology. Nanotechnology is presently applied in electrical engineering, chemistry, material science, cosmetics and sunscreens. The word nanoparticle is used to define a particle with size in the range of 1–100 nm. Atoms with size less than 100 nm are measured due to their unusual properties, such as low melting point, unique optical properties, high catalytic activity, and mechanical properties. Medicinal sciences are investigating the use of nanotechnology to improve medical diagnosis and treatments [1-2]. Metal oxide Nanoparticles, nanotubes, nanorods, nanowires, and whiskers are finding important applications which have been ranging from catalysis to optoelectronics. Nanomaterials behave differently as the size changes with respect to the bulk [3-4]. NPs are of great interest due to their extremely small size and large surface-to-volume ratio, which leads to both chemical and physical differences in their properties (e.g., Mechanical, biological, catalytic activity, thermal and electrical conductivity, optical absorption, and melting point) that has been compared to the bulk of the same chemical composition [5-6].

ZnO nanoparticles

ZnO is a multitasking metal oxide [7]. Compared to other 1-D semiconductors, ZnO has received its popularity as ZnO is a unique material with a direct band gap (3.37 eV) and large exciton binding energy of 60 meV [8]. The presence of semiconductor metal zinc oxide NPs (< 30 nm) in the biological system has ability to altered biological properties [9]. It has been widely used in UV absorbers and as an additive in many industrial products [10]. ZnO NPs have found applications in sunscreens, paints, and coatings as they are transparent to visible light and offer high UV absorption and are also being used as an ingredient in antibacterial creams, ointments and lotions, self-cleaning glass, ceramics, and deodorants. ZnO NPs offered preservatory effects on the food material [11].

ZnO NPs have also been used as an anti-microbial preservative for food products [12]. ZnO NPs showed effective antibacterial activity against Gram negative and Gram-positive bacteria [13]. ZnO exhibits very good photochemical reactivity and efficiently degrades the toxic water pollutants released from textile and dying industries by utilizing natural source of energy, sunlight. This is due to the presence of many active sites and

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fabrication of hydroxyl radicals on the ZnO surface [14]. Further, the Zinc oxide is expected to be an alternating material that can be synthesized into different shapes and sizes very easily [15].

Ag doped ZnO nanoparticles

ZnO samples were doped with a variety of dopants, changing the orientation and morphology either through heat treatment or by varying the concentration and dopant types. Ag-doped ZnO is suitable for transparent and optoelectronic-related technical applications due to its good stability and compatibility with other transparent conducting oxide materials at high temperatures. Ag is preferred to achieve improved luminescence efficiency. To date, various methods have been employed to create doped ZnO nanoparticles. Ag-doped ZnO nanocomposites may be used in chemical and biological sensors to control bacterial growth in a variety of applications, including medical devices, dental composite materials, and textile materials [16].

Due to the increased demand for various metallic and non-metallic NPs over the past two decades, a wide range of physical and chemical techniques have been developed to produce NPs of different sizes, shapes, and compositions [17]. Various physical, chemical and biological synthesis methods have been followed for obtaining these metal oxide NPs and among them chemical synthetic route is widely accepted which uses a range of inorganic and organic reducing agents. Chemical synthesis methods include emulsion solvent extraction method, double emulsion and evaporation method, salting out method, emulsion diffusion method, and solvent displacement/precipitation method. However, this method has many disadvantages like expensive, toxic, high pressure and energy requirement, difficult to separation and highly dangerous [18]. Therefore, biologically synthesized nanomaterial has become an important branch of nanotechnology [19]. The synthesis of NPs via eco-friendly routes have become popular among researchers due to its low cost, synthesis in ambient atmosphere, non-toxicity, environmental compatibility, etc. and ease of applications as the resulting particles are highly soluble in water, biocompatible, and devoid of toxic stabilizers [20]. More and more researchers are, therefore, now integrating green chemistry principles into their synthetic approaches for green nano products. Increasing awareness towards green chemistry has led to the development of an eco-friendly approach for the synthesis of metal oxide NPs [21]. The use of agricultural wastes or plants and their parts has emerged as an alternative to chemical synthetic procedures because it does not require

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elaborate processes such as intracellular synthesis and multiple purification steps or the maintenance of microbial cell cultures [22].

Aegle marmelos

Aegle marmelos also called as Indian bael has various medicinal values and traditional beliefs. It is called by different names over different parts of India such as bilva, bel, sadaphal and shriphal., It is used in the worship of Lord Shiva from historic times and hence commonly known as 'Shivadurme', the tree of Shiva. *Aegle marmelos* mainly contain terpenoids and phenolic compositions; this acts as a potential antioxidant and reducing agent [23-24]. The *Aegle marmelos* is one of the traditional plants belongs to Rutaceae family and have been used to cure ophthalmia, deafness, aggravations, catarrh, diabetes, and asthmatic dissensions. The natural products are utilized as a part of treating loose bowels, stomach hurt, and cardiovascular afflictions [25].

2. Literature Review

In order to understand the concept of green synthesis of NPs from plant extracts, a brief review of literature has been presented in the following paragraphs.

Atul *et al.,* (2009) reported Microwave assisted synthesis of ZnO: Cu nano-phosphors and their photoluminescence behaviour. Doped ZnO nanophosphors were characterized using X-ray diffraction analysis, transmission electron microscopy, steady state photoluminescence and time resolved laser induced photoluminescence spectroscopy. XRD patterns were confirming Wurtzite crystal of the synthesized Cu doped nanospheres geometry and TEM observations confirmed the formation of various nanostructures and their morphology. Photoluminescence emission spectrum observed shows the several transitions in visible region. [26].

Talam *et al.,* (2012) proposed a method for Synthesis, Characterization, and Spectroscopic Properties of ZnO Nanoparticles. ZnO nanoparticles have been synthesized by precipitation method from Zinc nitrate. XRD patterns showed that ZnO nanoparticles have hexagonal unit cell structure. The UV-vis absorption spectrum shows an absorption band at 355 nm due to ZnO nanoparticles. The photoluminescence spectrum exhibits two emission peaks one at 392 nm corresponding to band gap excitonic emission and another located at 520 nm due to the presence of singly ionized oxygen vacancies. [27].

Ahmed A. *et al.,* (2013) reported the Novel hexagonal two-dimensional ZnO nanosheets successfully and economically synthesized using zinc acetate and urea based on a facile

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microwave hydrothermal method. Optical properties of ZnO nanosheets were investigated by UV-Vis absorption and photoluminescence (PL) techniques [28].

Ambika *et al.*, (2015) reported Bio-mediated synthesis of metal oxide nanoparticles using plant extract is a promising alternative of traditional chemical synthesis. This study reports the synthesis of ZnO nanoparticles by biological method. The Highly stable and hexagonal phase ZnO nanoparticles were synthesized by using *Pongamia pinnata* leaves extract [29].

Suresh *et al.*, (2015) proposed a facile green synthesis method for Zinc oxide nanoparticles (ZnO Nps) using aqueous Extract of Chironji (*Buchanania lanzan*) leaves as fuel by solution combustion method. The Nps were evaluated for photoluminescence, photodegradation, antimicrobial and antioxidant activities. The extract was found to be rich in anti-Oxidants with 26% polyphenols and 25% flavonoids [30].

Zhang *et al.*, (2015) studied Photoluminescent ZnO Nanoparticles and Their Biological Applications. The photoluminescence of ZnO NPs usually contains two parts, the exciton-related ultraviolet (UV) emission and the defect-related visible emission. With respect to the visible emission, many routes have been developed to synthesize and functionalize ZnO NPs for the applications in detecting metal ions and biomolecules, biological fluorescence imaging, nonlinear multiphoton imaging, and fluorescence lifetime imaging [32].

Mahmodi *et al.*, (2018) proposed a method for the Cd selenide quantum dots ZnO nanocomposite: Synthesis, Characterization, dye removal ability with UV irradiation and antibacterial activity as a safe. Cd se quantum dots and ZnO nanocomposites synthesized and characterized its photocatalytic degradation ability was investigated [33].

Alzahrani *et al.*, (2018) proposed a method of chitosan embedded with ZnO/CuO nanocomposites for photodegradation of fast green dye under artificial and solar irradiation. The purpose was to degrade FCF. Thin film chitosan membrane was filled with ZnO nanocomposites and characterized by XRD, EDX, SEM [34].

Suresh *et al.*, (2019) proposed a method for Biosynthesis of nanocrystalline Zn-Mg-Cu oxide nanocomposite and their antimicrobial behavior. The mixed oxides nanocomposites were characterized by using XRD SEM EDX analysis. The biosynthesized oxides have shown great antimicrobial activity under laboratory condition [35].

Dahman et al., (2020) reported Pure and Mg-doped ZnO nanoparticles based to sol–gel route under supercritical conditions of ethyl alcohol. The samples were characterized by

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certain techniques. XRD patterns showed the hexagonal wurtzite crystalline structure of ZnO and Mg-doped ZnO nanoparticles. The TEM image demonstrates the hexagonal shape and the nanometric size of the nanostructures. SEM images show the presence of hexagonal like shape nanoparticles and the increase of grain size for the Mg doped synthesized sample [36].

Joseph *et al.*, (2021) proposed a method for Microwave assisted green synthesis of silver nanoparticles for optical, catalytic, biological and electrochemical applications. A novel and stable silver nanoparticle using *Cyanthillium cinereum* act as a reducing as well as capping agent by rapid microwave-assisted green method [37].

Ahmed *et al.*, (2021) studied Eco-friendly green synthesis approach has been employed for the formation of ZnO and Ag-doped ZnO nanoparticles using the aqueous extract of *Azadirachta indica* (Neem) leaves as reducing agent. Surface morphology was evaluated with scanning electron microscopy while crystalline size confirmed by XRD [38].

Rajini verma *et al.*, (2021) reported ZNO nanomaterials: green synthesis, toxicity evaluation and biomedical applications. ZnO nanomaterial are most widely used material in health care and environmental remedial applications lead to biodegradability [39].

Zhang *et al.*, (2021) reported green mechanism for the synthesis of ZnO NPs using the extracts of plants, exploring factors that influence the morphology of ZnO NPs and their antibacterial properties, and the mechanisms of antibacterial action. The results indicate that the factors that influence morphology include the intrinsic crystallographic morphological properties and conditions of the preparation of ZnO NPs [40].

Raghu et al., (2022) reported synthesis of ZnO NPs from *Synadium grantii* leaf extricate with varying Cu-dopant concentrations. In order to the synthesis of the pure and Cu-doped ZnO NPs, zinc nitrate hexahydrate and copper nitrate trihydrate were used as a precursor in the synthesis of nanoparticle using leaf extracts of the plant. XRD, TEM, FTIR, XPS, and PL measurements were carried out to examine the physical and optical properties of the synthesized samples. The photocatalytic studies of the prepared samples were studied using Methylene blue (MB), Indigo Carmine (IC), and Rhodamine B (RhB) organic pollutants. The wurtzite crystal structure of synthesized samples was confirmed by XRD and TEM analysis [41].

3. Research Gap Identification

Infact many Researchers have studied synthesis of CuO, FeO, NiO nanoparticles by different synthesis methods. ZnO Nanoparticles have been synthesized by many

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researchers mostly by chemical methods and green synthesis methods but there is very less literature done on the synthesis of ZnO nanoparticles by microwave assisted green synthesis method. Therefore, in this work we have decided to synthesize ZnO and Ag doped ZnO nanoparticles using *Aegle mermelos* via Microwave assisted green synthesis method.

4. Objectives

The main aim is to synthesize ZnO nanoparticles using *Aegle marmelos* and Mint leaf extract:

A) To synthesize and characterize ZnO and Ag doped ZnO nanoparticles.
B) To study antibacterial activity of synthesized ZnO and Ag doped ZnO nanoparticles.
C) To study photocatalytic activity of synthesized ZnO and Ag doped ZnO nanoparticles
D) To study antioxidant activity of synthesized ZnO and Ag doped ZnO nanoparticles.

5. Proposed Methodology

Material Required

Material required for synthesis of ZnO and Ag doped ZnO nanoparticles are *Aegle marmelos* (Bael), Mint leaves, Zinc Nitrate hexahydrate, silver nitrate and distilled water.

Apparatus required

Apparatus used for synthesis of ZnO and Ag doped ZnO nanoparticles are Microwave oven, measuring cylinder, Stirrer beaker.

Experimental procedure

Synthesis of ZnO nanoparticles using *Aegle marmelos* leaf extract

Aegle marmelos (bael) leaves will be collected from nearby area of Hamirpur Himachal Pradesh. Leaves will be removed from tree. Then leaves will be washed properly with water and dried. The leaves will be crushed and made into extract. Then Zinc Nitrate Hexahydrate will be dissolved to varying volume of extract. Stir the mixture using glass rod until the mixture will be homogenized. After this mixture will be kept in the microwave oven for about 5-10 minutes. The sample has been taken from oven and washed with ethanol/ water. Sample were dried in oven Creamish ZnO nanoparticles were obtained.

Synthesis of Ag -doped ZnO nanoparticles Using *Aegle marmelos* leaf extract

Aegle marmelos (bael) leaves will be collected from tree at nearby area of Hamirpur Himachal Pradesh. Leaves will be removed from tree. Then leaves will be washed

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properly with water and dried. The leaves will be crushed and made into extract. Then dopant silver nitrate along with Zinc Nitrate Hexahydrate will be dissolved to varying volume of extract. Stir the mixture using glass rod until the mixture will be homogenized. After this mixture will be kept in the microwave oven for about 5-10 minutes. Sample has been taken out from oven and Ag doped ZnO nanoparticles were obtained.

6. Characterization of ZnO and Ag doped ZnO nanoparticles

Ultra-Violet (UV) Spectroscopy

UV Visible spectroscopy is an analytical technique that measures the amount of wavelength of UV or Visible light that are absorbed or transmitted through a sample. This property is influenced by the sample composition, providing information about what is in the sample and at what concentration. UV spectroscopy information may be presented as a graph of absorbance, optical density or transmittance as a function of wavelength. [42]. UV Visible spectroscopy can be used to monitor the progress of reaction as well as detect impurities in a sample.

X-Ray Diffraction (XRD)

XRD is an important method to characterize the structure of crystalline material, lattice parameters, arrangement of individual atoms in single phase of polycrystalline material. XRD techniques have a number of advantages in forensic work and in the examination of documents; they are rapid, non-destructive and applicable to both very large and very small samples. In many cases the minerals used as filters in paper-making may also are identified by XRD. A certain amount of the information obtained by XRD can also be obtained by elemental analysis.

Transmission Electron Microscopy (TEM)

Optical microscopy and scanning electron microscopy are common techniques in mineralogical and structural investigations of geological and archaeological samples. The more complex and advanced transmission electron microscopy so far has been rarely used in geological and archaeological research. TEM is used together with other analytical methods in a comparative study of ancient pottery to identify copper- and iron-based pigments, to characterize the microstructures of opaque glass mosaics from a mediaeval church in Italy. A promising area of application for TEM, important to geology and archaeology, is to products of combustion [43].

Fourier-Transform Infrared (FTIR) Spectroscopy

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Fourier transform infrared spectroscopy (FTIR) is a technique used to obtain an absorption or emission infrared spectrum of a solid, liquid or gas. An FTIR spectrometer simultaneously collects high spectral resolution data over a wide spectral range. This technique is an established tool for the characterization of the samples [44].

Energy-Dispersive X-Ray Spectroscopy

This spectroscopy is used to identify elements of unknown composition. The emission spectra of molecules can be used in the chemical analysis of substances. This technique is also called as EDS.

Thesis Outline

Chapter 1 (Introduction): This chapter will include the detail about ZnO and Ag doped ZnO nanoparticles.

Chapter 2 (Literature Review): This chapter will deal with the intense literature survey of ZnO and Ag doped ZnO nanoparticles.

Chapter 3 (Experimental and Characterization Techniques): This chapter will describe the material and apparatus used for synthesis of ZnO nanoparticles and Ag doped ZnO nanoparticles and in this chapter various techniques and methods have been discussed which are used for the confirmation of the Zinc oxide nanoparticles and Ag doped ZnO nanoparticles.

Chapter 4 (Result and Discussion): This chapter will describe detail discussion on results of present work which includes synthesis of ZnO and and Ag doped ZnO nanoparticles the applications of these synthesized quantum dots.

Chapter 5 (Conclusion): This chapter will comprise summary of entire study according to objectives along with conclusions.

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CERTIFICATE
(SYNOPSIS APPROVAL CERTIFICATE)

This is to certify that the Revised Synopsis for Ph.D. Thesis titled, **"MICROWAVE ASSISTED GREEN SYNTHESIS OF ZNO AND AG-DOPED ZNO NANOPARTICLES USING AEGLE MARMELOS LEAF EXTRACT"**, submitted by **Ms. SAKSHI DHIMAN** to the Career Point University, Hamirpur (H.P.), India in partial fulfillment of the requirements for the degree of **DOCTOR OF PHILOSOPHY** in the Division/Department of Chemistry, School of **Basic and Applied Sciences** has been approved by the Research Advisory Committee (RAC) after an Oral Examination of the student on dated 17.06.2023.

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